

10/496,773

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=> s sterically(1)hindered(1)amine
    16257 STERICALLY
    36916 HINDERED
    266015 AMINE
L1      659 STERICALLY(L)HINDERED(L)AMINE

=> s organic(1)peroxide
    352483 ORGANIC
    201533 PEROXIDE
L2      1588 ORGANIC(L)PEROXIDE

=> s iodide or bormide or chloride
    172600 IODIDE
        4 BORMIDE
    1074880 CHLORIDE
L3      1201656 IODIDE OR BORMIDE OR CHLORIDE
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10/496,773

=> s 11 and 12
L4 0 L1 AND L2

=> s 12 and 13
L5 122 L2 AND L3

=> s 15 and 11
L6 0 L5 AND L1

=> s 15 and amine
266015 AMINE
L7 5 L5 AND AMINE

=> d 17 1-5 bib abs

L7 ANSWER 1 OF 5 CAPLUS COPYRIGHT 2006 ACS on STN
AN 2004:965634 CAPLUS
DN 142:113417
TI Effective and selective iodofunctionalisation of organic
molecules in water using the iodine-hydrogen peroxide tandem
AU Jereb, Marjan; Zupan, Marko; Stavber, Stojan
CS Department of Chemistry, Faculty of Chemistry and Chemical Technology,
University of Ljubljana, Ljubljana, 1000, Slovenia
SO Chemical Communications (Cambridge, United Kingdom) (2004), (22),
2614-2615
CODEN: CHCOFS; ISSN: 1359-7345
PB Royal Society of Chemistry
DT Journal
LA English
OS CASREACT 142:113417
AB Efficient hydrogen peroxide enhanced iodofunctionalization of ketones,
1,3-dicarbonyl compds. and activated aromatic mols. using elemental iodine in
water is achieved, whereas alkynes were stereoselectively converted into
(E)-1,2-diiodoalkenes.

RE.CNT 20 THERE ARE 20 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L7 ANSWER 2 OF 5 CAPLUS COPYRIGHT 2006 ACS on STN
AN 2001:242083 CAPLUS
DN 135:25078
TI Deposition of Titania Thin Films by a Peroxide Route on
Different Functionalized Organic Self-Assembled Monolayers
AU Niesen, Thomas P.; Bill, Joachim; Aldinger, Fritz
CS Max-Planck-Institut fuer Metallforschung und Institut fuer
Nichtmetallische Anorganische Materialien, Universitaet Stuttgart
Pulvermetallurgisches Laboratorium, Stuttgart, D-70569, Germany
SO Chemistry of Materials (2001), 13(5), 1552-1559
CODEN: CMATEX; ISSN: 0897-4756
PB American Chemical Society
DT Journal
LA English
AB Self-assembled monolayers (SAMs) on single-crystal Si wafers were used as
substrates for the deposition of oxide thin films. The organic surface is
effective for promoting the growth of films from aqueous solns. at temps.
<100°. The formation of a Ti complex in the presence of H2O2 is
used to stabilize an otherwise spontaneously precipitating aqueous Ti solution
Uniform
TiO2 films were formed at 80° on sulfonated SAMs, whereas hydroxyl
and amine functionalities led to inhomogeneous coatings. The
films were characterized by a variety of techniques, including
ellipsometry, RBS, AFM, SEM, and TEM, to determine the thickness, topog.,
microstructure, and chemical composition of the films. An electronegativity-pH

diagram is used to explain how the composition of the deposition solution is reflected in the final thin film, both for the present films and for TiO₂ films reported earlier in the literature. Finally, the current understanding of the film formation mechanism is discussed.

RE.CNT 45 THERE ARE 45 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L7 ANSWER 3 OF 5 CAPLUS COPYRIGHT 2006 ACS on STN
AN 1966:4076 CAPLUS
DN 64:4076
OREF 64:702b-h
TI 4H-Benzo[4,5]cyclohepta[1,2-b]thiophene derivatives
PA Sandoz Ltd.
SO 9 pp.; Addn. to Neth. Appl. 6,408,529 (CA 63, 4264c)
DT Patent
LA Unavailable
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	NL 6414606		19650621	NL 1964-14606	19641215
PRAI	CH		19631220		

GI For diagram(s), see printed CA Issue.

AB The title compds. which show neuroleptic and antidepressive properties were prepared through hydrolysis and dehydration of the product of a Grignard reaction of 4H-benzo[4,5]cyclohepta[1,2-b]thiophen-4-one (I) with a halo amine. Thus, a few drops EtBr were added to 2.2 g. iodine-activated Mg in 15 ml. absolute tetrahydrofuran (THF) and a solution of 10.8 g. 3-dimethylaminopropyl chloride in 20 ml. absolute THF was added at such a rate to keep the solution boiling, the refluxing was continued 1 hr. after the addition, a solution of 8.2 g. I in 30 ml. THF was then added over 15 min. at 20° and the whole refluxed 30 min. The cooled mixture was poured into 250 ml. saturated aqueous NH₄Cl, 200 ml. CH₂Cl₂

added

and the whole filtered over diatomaceous earth, the aqueous layer extracted with

CH₂Cl₂, the combined exts. were washed with H₂O, dried, and evaporated under 15 mm. to give a residue, which, recrystd. in EtOH-hexane gave 4-(3-dimethylaminopropyl)-4H-benzo[4,5]cyclohepta[1,2-b]thiophen-4-ol (II), m. 121-2°. A solution of 4 g. II in 250 ml. Ac₂O was refluxed 6 hrs. after which 200 ml. solvent were evaporated and the residue poured in 1200 ml. H₂O, the aqueous layer was filtered over diatomaceous earth, and extracted with Et₂O after basification with 20% NaOH. The washed and dried extract was evaporated to give 4-(3-dimethylaminopropylidene)-4H-benzo[4,5]cyclohepta[1,2-b]thiophene (III, R = 3-dimethylaminopropylidene), b_{0.1} 160-5°; malonate m. 142-3°. Thienyl chloride (133 g.) was stirred and heated at 150° and 175 g. PO₃Et₃ added dropwise and then stirred and heated at 160-70° for another 2 hrs. The mixture was distilled to give di-Et 2-thenylphosphonate (IV), b_{0.06} 120-4°. Dry MeONa (30 g.) was added to a solution of 117 g. IV in 200 ml. dimethylformamide (DMF), the temperature was raised to 45-50°, a solution of 80 g. o-phthalaldehydic acid in 200 ml. was added to the cooled mixture at such a rate to keep the temperature below 35-40°, the stirring was continued 30-60 min. at room temperature and the whole was then poured on 1600 ml. H₂O at 10-5°; a red oil separated but was dissolved again when the solution was basified with K₂CO₃, the brown solution was extracted with C₆H₆, the aqueous

layer was acidified to pH 4 at 10-15° with HCl and cooled to give 2-[2-(2-thienyl)vinyl] benzoic acid (V), m. 133-5°. Na (7.5 g.) was amalgamated with 375 g. Hg and a solution of 20 g. V in 150 ml. 95% EtOH was added, the whole was shaken 30 min., the Hg was separated and washed with EtOH, 1600 ml. H₂O was added to the combined EtOH solns., after filtration over diatomaceous earth, the solution was acidified with HCl and cooled to

5° to give 2-[2-(2-thienyl)ethyl]benzoic acid, (VI), m. 110-11°. VI (20 g.) were added over 30 min. at 125-30° to a mixture of 86 g. P2O5 and 54 ml. 84% H3PO4, the whole was stirred 2 hrs. at 125-30° and then poured on 1000 ml. H2O, filtered over diatomaceous earth, and extracted with CH2Cl2, the organic layer was washed with 2N Na2CO3, dried, and evaporated to give 9,10-dihydro-4H-benzo[4,5]cyclohepta[1,2-b]thiophen-4-one (VII), b0.05 125-40°, n24D 1.6559. A mixture of 32.1 g. VII, 26.7 g. N-bromosuccinimide, and 0.3 g. benzoyl peroxide in 250 ml. dry CCl4 was refluxed 4 hrs., the solvent was evaporated under 15 mm. after being cooled and filtered over diatomaceous earth, the residue was heated 2 hrs. in the presence of 200 ml. Et3N, the excess amine was evaporated, and the residue dissolved in 250 ml. CH2Cl2, which, after being washed with 2N HCl and H2O, was dried and evaporated to give I, m. 109-10°, b0.1 173-80°. I and 2-(1-methyl-2-piperidyl)ethyl chloride gave III (R = 2-(1-methyl-2-piperidyl)ethylidene); salicylate m. 167-8°.

L7 ANSWER 4 OF 5 CAPLUS COPYRIGHT 2006 ACS on STN
AN 1962:73336 CAPLUS
DN 56:73336

OREF 56:14178h-i,14179a-h

TI Organic sulfur compounds. V. Alkylammonium thiolate and peroxide salts; possible intermediates in amine

-catalyzed oxidation of mercaptans by hydroperoxides

AU Oswald, Alexis A.; Noel, Fernand; Stephenson, Arthur J.

CS Imp. Oil Co., Sarnia, Can.

SO Journal of Organic Chemistry (1961), 26, 3969-74

CODEN: JOCEAH; ISSN: 0022-3263

DT Journal

LA Unavailable

AB cf. CA 56, 9994a. Aliphatic amines catalyzed the reaction of mercaptans and hydroperoxides to yield the corresponding disulfides, alcs., and H2O. The formation of alkylammonium salts of mercaptans and hydroperoxides was studied as a possible explanation of this catalysis. Alkylammonium peroxides formed a new class of hydroperoxide salts. They reacted with aromatic mercaptans at a very fast rate to yield alkylamines, plus the same products as the amine catalyzed mercaptan oxidation by hydroperoxide. 1,1,3,3-Tetramethylbutylammonium 2-naphthalenethiolate (I) (15 g.) in 290 ml. PhMe was treated with 2.2 g. tert-butyl hydroperoxide in 250 ml. PhMe, the mixture saturated with dry HCl, concentrated to 200 ml.

in vacuo,

and from the concentrate 1,1,3,3-tetramethylbutylammonium chloride (II) extracted by 2 100-ml. portions of 5% HCl. Filtration and recryst. gave 4.4 g. bis-2-naphthyl disulfide, m. 139-40°. The combined aqueous HCl fractions yielded 5.9 g. II, m. 230-6°, which (treated with 2,4-dinitrobenzenesulfenyl chloride) gave 2,4-dinitrobenzenesulfenyl-N-(1,1,3,3-tetramethyl)butylamide, m. 103-4°. Analysis by partition chromatography of another sample of the original mixture showed that about 85% of the calculated Me3COH concentration was present. Karl Fischer H2O determination gave 105% of the theoretical amount of H2O. About 0.2 g. of the peroxide in 20 ml. C6H6 containing 0.25 mole/l. 2-naphthalenethiol was treated with about 0.1 ml. 1,1,3,3-tetramethylbutylamine (III), the mixture left 0.5 hr., and an aliquot titrated for thiol content with AgNO3 (using a potentiometer with an Ag-glass electrode pair). The reaction between the thiol and peroxide was usually completed in a few min. in the presence of III (as was indicated by the yellow coloration of the solution). Analyses of some hydroperoxide samples gave the values as shown: (method, % tert-Bu, phenylisopropyl, and tetrahydronaphthyl hydroperoxide given): mercaptan, 73, 65, 99; iodide, 74, 63, 101. The thiol (0.05 mole) was added dropwise to 50 ml. ethereal solution of 0.05 mole aliphatic amine and the precipitate filtered off, washed, and dried 0.5 hr. at 10 mm. I (13 g.) in 4 l. 3:1 C6H6-heptane oxygenated 6 hrs., then concentrated in

vacuo, HCl passed in, and the product crystallized gave 6.6 g. II, identified as 1,1,3,3-tetramethylbutylammonium picrate, m. 177-80°. The amine content of the aerated reaction mixture, determined by extraction with aqueous HCl, was 5.3 g. When a solution of 0.84 g. 2-naphthalenethiol in 100

ml.

3:1 C6H6-heptane was aerated in the absence of amine, the thiol concentration decreased to 87% of the original. A hydrocarbon hydroperoxide (0.05 mole) added to 2.8 g. diazabicyclo[2.2.2]octane in 50 ml. Et2O, the solution cooled, and the product removed, washed, and dried gave the triethylenediammonium alkyl peroxide. These peroxides could also be synthesized in hydrocarbon solvents or in the absence of any solvent. PhMe and ligroine were suitable. The following N-substituted ammonium alkyl peroxides were thus obtained (N compound, moles, hydroperoxide, moles, m.p. of the product, solvent, and % yield given): C16H33NH2, 2, Me2CMe2(CH2)2CMe2O2H (IV), 1, 50-6°, PhMe, 73; N(CH2CH2)3N, 1, Me3CO2H, 2, 74-6°, Et2O, 93; N(CH2CH2)3N, 1, PhCMe2O2H, 1, 87.5-8.5°, Et2O, 96; N(CH2CH2)3N, 1, α -naphthylhydroperoxide, 2, 47-8°, Et2O, 91; MeN(CH2CH2)2NMe, 1, IV, 1, semisolid, PhMe, 86; N(CH2CH2)3N, 1, IV, 1, 128-31°, Et2O, 82; HN(CH2CH2)2NH, 1, IV, 2, 74-5°, PhMe, 88; N(CH2CH2)3N, 1, Me3CO2H, 1, 52-5°, Et2O, 90; hexamethylenetetramine (V), 1, Me3CO2H, 2, 74° (decomposition), -, 52; V, 2, IV, 5, 88-90°, PhMe, 8; acridine, 2, IV, 1, 104-5°, Et2O, 92, phenazine, 1, IV, 1, 90° (decomposition), PhMe, 61. The following alkylammonium thiolates were prepared (salt of -amine, -thiol, m.p. of product, and % yield given): dodecyl (VI), 4-toluene, 89-90.5°, 84; VI, 2-naphthalene (VII), 94-5.5°, 83; VI, 4-chlorobenzene, 87-8.5°, 97; decyl (VIII), benzene (IX), 87.5-9.0°, 85; VIII, VII, 87-8.5°, 87; 2-hydroxyethyl, VII, 84-6°, 75; octyl (X), IX, 83-4°, 96; X, VII, 112-14°, 86; cyclohexyl (XI), IX, 122-5°, 84; XI, phenylmethane, 56-8°, 85; tris(2-hydroxyethyl), VII, 72-5°, 98; triethylenedi, IX, 96.5-8.5°, 88. Characteristic infrared absorption peaks of alkylammonium hydroperoxides were given in a table.

L7 ANSWER 5 OF 5 CAPLUS COPYRIGHT 2006 ACS on STN

AN 1917:15617 CAPLUS

DN 11:15617

OREF 11:3130b-e

TI Vulcanizing rubber

IN Hopkinson, E.

DT Patent

LA Unavailable

FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 108453		19161208	GB	

PI Natural or synthetic rubber is vulcanized, with or without the aid of S, by means of benzoyl peroxide, perbenzoic acid, and the like, ozonide of rubber, rubber chlorides, bromides, and hydrochlorides, or chlorides and bromides of synthetic rubbers, dimethylethylene rubber bromide, cauprene chloride or bromide, trinitrobenzene, nitro-organic dye-stuffs, e. g., erythrosin, eosin, alkali blue, and cinnamylidene-fluorene. The claims comprise also the employment of peroxides, peracids, a rubber compound adapted to produce vulcanization, and S-free organic dyes containing O. Rubber is vulcanized by means of mixts., such as S, trinitrobenzene, and PbO with or without naphthylamine; S, PbO, and piperidine piperidylthiocarbamate or in place of the latter isoamylamine, hexamethylenetetramine, hexamethylenetetramine dithiocarbamate, or other aliphatic amine; and S, ZnO, erythrosin, and piperidine piperidylthiocarbamate. In some cases, a temperature

above 100° is necessary for vulcanization, whereas in others it may be carried out below this temperature or even at normal temperature. Rubber ozonide,

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referred to above, may be prepared by treating layers of rubber from 0.5 to 1 mm. thick with a stream of dry air under the influence of rays from a Hg lamp. When the weight has increased by 0.2 to 1%, the product is milled between cold rollers and reheated for 1-15 min. at 100-120°. The halogen derivs. of rubber referred to may be prepared by the direct action of halogens on solns. of natural and synthetic rubbers.

=> log y

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